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AD-A035 136

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Picatinny Arsenal, Dover, New Jersey

January 1977

AD A0

TECHNICAL MEMORANDUM 2384

COMPATIBILITY OF FLOCCULATING AGENTS WITH RDX/TNT/COMP B

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JANUARY 1977

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REPORT DOCUMENTATION PAGE

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1. REPORT NUMBER Technical Memorandum 2224	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) COMPATIBILITY OF FLOCCULATING AGENTS WITH RDX/TNT/COMP B		5. TYPE OF REPORT & PERIOD COVERED Picatinny Arsenal Technical Memorandum 6. PERFORMING ORG. REPORT NUMBER Explosives Div, FRL
7. AUTHOR(s) Thomas C. Castorina James W. McCahill Arthur C. Forsyth		8. CONTRACT OR GRANT NUMBER(s) 5084-01-001
9. PERFORMING ORGANIZATION NAME AND ADDRESS Explosives Division, FRL Picatinny Arsenal, Dover, New Jersey 07801		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS Reimbursable Services TEAD-05-76 (11 Aug 1975)
11. CONTROLLING OFFICE NAME AND ADDRESS Tooele Army Ammunition Depot Tooele, Utah 84074		12. REPORT DATE January 1977 13. NUMBER OF PAGES 14
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

from pg 1

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Cationic polyelectrolytes
Polyacrylamides
Coagulants
100°C vacuum stability test
Energetic materials

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Of the flocculating agents, WT2600, CAT-FLOC, CAT-FLOC-T, and E-653, tested for their compatibility with RDX, TNT and Comp B, only E-653 was found to be acceptable. A possible mechanism is proposed for the reactivity of the polymer flocculating agents with explosives.

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ACKNOWLEDGEMENTS

The authors wish to thank Mr. Allan Parkinson of Tooele Army Ammunition Depot, Tooele, Utah for his support on this project; Dr. Robert Slagel of Calgon Corporation for providing the polymer samples; and Mr. James Murphy of the Propellants Research Branch for the vacuum stability test data.

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INTRODUCTION

This study was undertaken at the request of the Tooele Army Ammunition Depot (TAAD), under Intra Army Order for Reimbursable Services TEAD-05-76 (11 Aug 1975), to determine the compatibility of polymer flocculating agents with explosives present in waste waters at Army Ammunition Plants. These agents are used effectively for the removal of the suspended matter (Ref 1), in aqueous media. As such, they could serve to remove colloidal suspensions of explosives which clog the charcoal filters that are used to adsorb solubilized explosives from waste water effluents (Ref 1). Before these flocculating agents can be accepted as standard items for the pre-treatment of waste water effluents at explosives processing plants, their compatibility with explosives in the dry state must be established. This report describes the various flocculating agents screened for this purpose. go to BDS pg 1

EXPERIMENTAL PROCEDURE

Compatibility Test

The standard Compatibility Test (Reactivity of 100°C Vacuum Stability Test) (Ref 2) was used to determine the compatibility of the explosives with the polymer flocculating agents selected for investigation. The samples, consisting of 2.5g of explosive, 2.5g of polymer, or 5g of a mixture, 50% explosive, 50% polymer by weight, were placed into a glass heating vial which was attached to a capillary tube. The assembly was then evacuated and sealed by the mercury column used to measure the pressure of the gases evolved. The vial was placed in a constant temperature block heater set at $100 \pm 0.5°$ for 40 hours and the volume of gas evolved was recorded. The difference between the volume evolved from the mixture and from the sum of the volumes of gases evolved separately from each of the components in the mixture was considered to be directly proportional to the instability or incompatibility of the mixture.

The rating table of compatibility is as follows (Ref 2):

ml. Excess Gas	Degree of Reactivity
0.0 – 3.0	Negligible
3.0 – 5.0	Moderate
5.0 and above	Excessive

Flocculating Agents

The polymeric flocculating agents tested were supplied by Calgon Corporation, Pittsburgh, PA with their respective descriptions:

1. WT-2635, a cationic homopolymer of ethenimine, clear to pale yellow viscous polyelectrolyte, completely water soluble (Fig 1).

2. WT-2600, a cationic copolymer of dimethyldiallyl ammonium chloride and acrylamide, off-white, flaked polyelectrolyte, completely soluble in water (Fig 2).

3. CAT-FLOC, a cationic homopolymer of dimethyldiallyl ammonium chloride polyelectrolyte, clear water-white to pale yellow viscous liquid (Fig 3).

4. CAT-FLOC-T, a liquid cationic polyelectrolyte, of identical chemical composition as CAT-FLOC except for its higher molecular weight distribution (Fig 3).

5. E-653, an experimental sulfonated polyacrylamide (Chemical structure proprietary) powder hydrolytically stable under acidic conditions at elevated temperatures and with acidic compounds, including explosives (Ref 3).

The flocculants received in liquid form were isolated as solids by mixing them with an equal volume of methanol and adding dropwise to ten times their volume of acetone with very rapid stirring. The stringy polymer which precipitated was shredded with scissors while under acetone, then vacuum dried at 20 to 25 inches of mercury at 55°C to constant weight (approximately 2 hours).

Explosives

The explosives examined for compatibility with the polymer flocculants in the solid state were:

RDX:	Lot HOL-SR4-57
TNT-B:	Batch Process, Lot VOL-7-897
TNT-C:	Continuous Process, Lot RAD-7-0220
Comp B:	Lot HOL-053-5034

Samples of TNT and Comp B were prepared for the compatibility test by dissolving 10-15 gram quantities in 10-15 ml of acetone. The solutions were added dropwise to

CATIONIC HOMOPOLYMER OF ETHLENIMINE

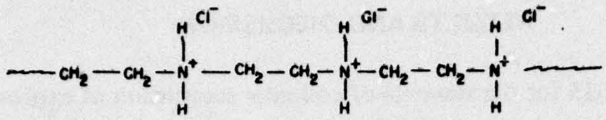

Fig 1 WT-2635

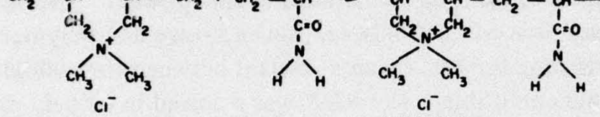

Fig 2 WT-2600

CATIONIC HOMOPOLYMER DIMETHYLDIALLYLAMMONIUM CHLORIDE

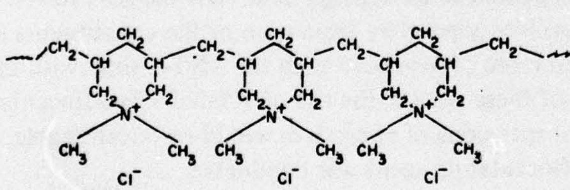

Fig 3 CAT-FLOC and CAT-FLOC-T

two liters of distilled water. The resulting fine precipitate was filtered and dried to constant weight.

RESULTS AND DISCUSSION

The use of WT-2635 for the removal of colloidal suspension of explosives was found by TAAD (Ref 3) to be very effective and hence prevented the clogging of the carbon filtration units in the pollution abatement system. However, before the use of WT-2635 could be standardized, its compatibility with the explosives involved had to be determined. The results of the compatibility tests are listed in Table 1.

It should be noted that the batch process TNT (TNT-B) was included with the continuous process TNT (TNT-C) in the test series because there are residual lots to TNT-B still available for processing at AAP's. The TNT and Comp B were transformed (see Experimental Procedure) from flakes/ chunks to fine powder. This was done to achieve maximum surface area contact between the explosive and polymer flocculant, approaching but not attaining the surface area contact between the colloidal explosives and polymer under actual conditions. The RDX was received in a finely divided crystalline form and used as such.

The compatibility test of the separate constituents which served as controls for the various mixtures showed that no significant background corrections were required. The RDX in combination with TNT-C and TNT-B, as well as with WT-2635 was stable. However, TNT-C and TNT-B mixed with WT-2635 failed the compatibility test, the former to a slightly lesser extent than the latter. When RDX was added to the TNT/polymer mixtures, the net result appeared to be synergistic in that the gas evolved was greater than the sum of the gas evolved separately from each of the constituents in the mixture. This synergistic effect was more pronounced with the TNT-C than with the TNT-B. As a preliminary evaluation of these results, the use of WT-2635 as a flocculating agent for the removal of colloidal suspensions of explosives would be questionable. Accordingly, a search for a substitute flocculating agent was conducted.

The polymer flocculants investigated for this purpose were WT-2600, CAT-FLOC and CAT-FLOC-T, supplied by Calgon Corporation, Pittsburgh, PA in sample quantities. The compatibility of these flocculants with RDX/TNT/COMP B, as determined by the vacuum stability test at 100°C is given in Table 2. RDX was observed to exhibit no reactivity with the three flocculating agents. Whereas, TNT-C and TNT-B were excessively reactive with WT-2600 and CAT-FLOC-T, but negligibly reactive with CAT-FLOC. However, when the TNT was combined with RDX (COMP B), a synergistically enhanced reactivity was observed with CAT-FLOC. The incompatibility of COMP B with

Table 1

Compatibility of WT-2635 with TNT/RDX/COMP B

Sample	Net ml gas/5 gm	Reactivity	Hours
WT-2635	0.11		40
TNT-C	0.00		40
TNT-B	0.00		40
RDX	0.12		40
RDX/TNT-C (60/40)	0.06	negligible	40
RDX/TNT-B (60/40)	0.05	negligible	40
WT-2635/TNT-C (50/50)	8.10	excessive	40
WT-2635/TNT-B (50/50)	11+	excessive	40
WT-2635/RDX (50/50)	0.65	negligible	40
WT-2635/TNT-C/RDX (50/30/20)	11+	excessive	20
WT-2635/TNT-B/RDX (50/30/20)	11+	excessive	16

Table 2

Compatibility of WT-2600/CAT-FLOC/CAT-FLOC-T with TNT/RDX/COMP B

Sample	Net ml gas/5 gm	Reactivity	Hours
WT-2600	0.90		40
CAT-FLOC	4.17		40
CAT-FLOC-T	1.74		40
RDX	0.13		40
TNT-C	0.17		40
TNT-B	0.28		40
COMP B	0.47		40
RDX/WT-2600	2.83	negligible	40
TNT-B/WT-2600	9.82	excessive	16
TNT-C/WT-2600	9.93	excessive	16
COMP B/WT-2600	9.63	excessive	16
RDX/CAT-FLOC	0.00	negligible	40
TNT-B/CAT-FLOC	0.74	negligible	40
TNT-C/CAT-FLOC	1.56	negligible	40
COMP B/CAT-FLOC	6.36	excessive	16
RDX/CAT-FLOC-T	1.87	negligible	40
TNT-B/CAT-FLOC-T	8.98	excessive	16
TNT-C/CAT-FLOC-T	9.09	excessive	19
COMP B/CAT-FLOC-T	8.79	excessive	16

WT-2600 and CAT-FLOC was consistent with the corresponding excessive reacitvity of TNT-C and TNT-B. In any case all three flocculating agents failed the compatibility test.

At this point an effort was made to gain some insight into the reaction mechanism causing the observed excessive reactivity between TNT/RDX and the polymeric flocculating agents. A computerized literature search was, therefore, conducted through PLASTEC at Picatinny Arsenal on the compatibility of TNT/RDX with polymers in general. From the conflicting data that was generated, some useful information was extracted, viz., that amino functional groups, such as in polyacrylamides, can exhibit reactivity with TNT. Examination of the chemical structures of the polymer flocculants tested, Figures 1, 2, and 3, show an imine and/or amine in their cationic form. Conceivably, on heating, the basic form interacts with TNT producing gaseous degradation products which in turn react with RDX. Based on such an assessment, a specific system involving an alternative reaction path was required if an acceptable flocculating agent was to be found. The polymeric flocculant had to be devoid of reactive cationic amines/imines or contain derivatized forms that would not interact with acidic compounds such as explosives.

A possible candidate flocculating agent meeting these requriements was an experimental anionic polymer, E-653, which was reportedly (Ref 3) hydrolytically stable under acidic conditions and, in particular, stable with acidic compounds. The chemical structure was declared proprietary at its present stage of development. However, it was disclosed that the polymer contained a derivatized form of polyacrylamide, i.e., a sulfonated polyacrylamide functional configuration. The acrylamide could be derivatized by one possible reaction path:

$$RCONH_2 + CH_2O + Na_2SO_3 \rightarrow RCONHCH_2SO_3^-Na^+$$

As such, the SO_3^- would serve to stabilize the amine functional group, thereby, preventing an ensuing basic interaction with acidic compounds. A complimentary sample of E-653 was sent by Calgon Corporation, upon request, for determining its compatibility with the explosives under investigation. The results of the compatibility test presented in Table 3 are self-explanatory and unequivocally illustrate what has been claimed for this polymer.

Table 3

Compatibility of E-653 with TNT/RDX/COMP B

Sample	Net ml gas/5 gm	Reactivity	Hours
RDX/E-653	0.00	negligible	40
TNT-B/E-653	0.00	negligible	40
TNT-C/E-653	0.08	negligible	40
COMP B/E-653	0.00	negligible	40

CONCLUSIONS

The acceptability of E-653 as a flocculating agent will depend upon how effective it is in precipitating colloidal suspensions of RDX/TNT. On the assumption that E-653 is just as effective in this respect and that it is, or will be equally as available as WT-2635, then a substitute polymer flocculating has been found for the removal of colloidally dispersed TNT and RDX in waste waters at LAP's. If there is still an interest in one of the flocculants which has been shown to be incompatible with the explosives, viz., WT-2635, WT-2600, CAT-FLOC and CAT-FLOC-T, then a procedure would be developed which would insure the safe handling of the flocculant-explosive slurry until its ultimate disposition. Information could be obtained on the safety of the dry mixture of the flocculant and the explosive by performing confined explosion temperature and impact sensitivity tests on the materials. An analysis of the DTA-TGA curves of the samples could also provide useful data. Impact sensitivity tests would help show the hazards, if any, in the handling of the mixture.

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